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THE RELATIONSHIP BETWEEN BALLISTIC
BEHAVIOR AND STRUCTURAL MICRODEFECTS OF T PROPELLANT
- AN APPLICATION OF SEM IN THE STUDY OF DOUBLEBASE PROPELLANT

by

Li Xuetong

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THE RELATIONSHIP BETWEEN BALLISTIC BEHAVIOR AND STRUCTURAL MICRODEFECTS OF T PROPELLANT - AN APPLICATION OF SEM IN THE STUDY OF DOUBLEBASE PROPELLANT

by Li Xuetong

Abstract

The micro-structure of a sampling T propellant at different ballistic behavior and under different manufacturing conditions is analyzed with a scanning electron microscope. The samples are taken from a homogeneous T propellant gelatinized by repellant solvent and shaped by thrusting. It is found that in the powder grain, a lot of structural microdefects are unevenly but regularly distributed; the defects on the samples of normal and abnormal ballistic behavior are clearly different; the grains with good structure made under specific manufacturing conditions show normal ballistic behavior and structural changes caused by low temperature are observed. On the basis of analyzing the micro-pictures, the mechanism of inherent structural defects of abnormal ballistic behavior for T propellant is presented and critical technology which makes the ballistic behavior normal is analyzed.

Fourteen SEM pictures are appended.

I. Preface

When certain propellants undergo combustion in a bore, they show abnormal low temperature ballistic behavior. A report [1]

made during the beginning ~~to~~ of the 1960's, indicated a useful sealed exploder simulated test to select a sieve which could examine the safety and danger of stored propellants. Research on the mechanical properties of propellants was not carried out under many conditions and so the reason why some people proposed the possibility of powder grain embrittlement was because the bore pressure rose [2]. Up to the present, much research has been done by scholars on the theory and testing of these mechnaical properties [3,4]. The brittle point of heterogeneous triple base propellant has also been given attention [5]. Most of this research begins with the effects of external forces on powder grains with different low temperature mechanical behavior (including ignition pressure and combustion shock, as well as the shock of the powder grain and base of the projectile and the shocks within the bore walls and in the powder grain) which produce and enlarge internal cracks and cause the bore pressure to rise abnormally.

This article considers that there is a close relationship between the mechanical properties of powder grain including embrittlement and ballistic behavior. Test results on mechanical properties indicate that there are many influencing factors for the macroscopic expression of the microscopic structural changes and after changing the test conditions, the scope was very large. The problem of how to effectively simulate powder grain in an unfavorable bore situation is complex. On the other hand, the random differences of the micro-structure influence mechanical strength. Yet, common tests of mechanical properties do not use real powder grain and thus this is the shortcoming of using mechanical properties to study the process within the bore.

When studying each powder grain within a solvent manufactured at different times but using similar methods and similar

thrusting, the maximum pressure under low temperature shows higher abormalities than the data for pressure under constant temperature. This article utilized a scanning electron microscope to observe the micro-structures of each powder grain and analyzes the conditions of the defects and the mechanism of the inherent defects of abnormal ballistic behavior for the propellant. Because there is a close relationship between the micro-structure and manufacturing technology, based on the principles of physical chemistry of the inherent defects formed in powder grains, the critical elements in manufacturing technology can be analyzed. Accordingly, the powder grain used in this analysis, which was manufactured by means of a special technological procedure, not only had good structure but tests also proved normal ballistic behavior.

II. Tests and Examples of Micro-Pictures

Each powder grain sample underwent graphite processing and had similar external appearances. As far as the section is concerned, it is light in color, it is a homogeneous material and there are no defects visible to the naked eye. Observations of symmetrically centered samples of powder grain cross sections were used to make semi-quantitative statistics on the defects, and the defects on the surface area were used for references for analysis. Based on the research goals, we can divide the test results into three groups.

The first group is concerned with the analysis of the micro-structures of fourteen T type powder grains which have similar manufacturing and technological methods and which show normal and abnormal ballistic behavior. T type-A group is a group with abnormal ballistic behavior and the T type-B group has normal ballistic behavior. Some of their typical visual

conditions and defect characteristics are listed in table 1.

(1) 序号	(2) 片号	(3) 样品批号	(4) 取样部位	(5) 倍率	(6) 缺陷特点
a	8247	(7) T型-B	(12) 中心	500×1/2	(17) Ⅰ级
b	8221	(8) T型-A	(13) 中心	500×1/2	(18) Ⅱ级
c	8239	(9) T型-A	(14) 中心	500×1/2	(19) Ⅲ级
d	8243	(10) T型-B	(15) 中心	500×1/2	(20) Ⅳ级
e	5765	(11) T型-A	(16) 非中心	500×1/2	(21) 定向

Table 1 Examples of the Classes and Directional Conditions of T Type Powder Grains

Key:
1. Sequence number
2. Picture number
3. Specimen group number
4. Sampling position
5. Multiplication rate
6. Characteristic of defect
7. T type-B
8. T type-A
9. T type-A
10. T type-B
11. T type-A
12. Center
13. Center
14. Center
15. Center
16. Not center
17. Class I
18. Class II
19. Class III
20. Class IV
21. Directional

See fig. 1 for pictures listed in table 1.

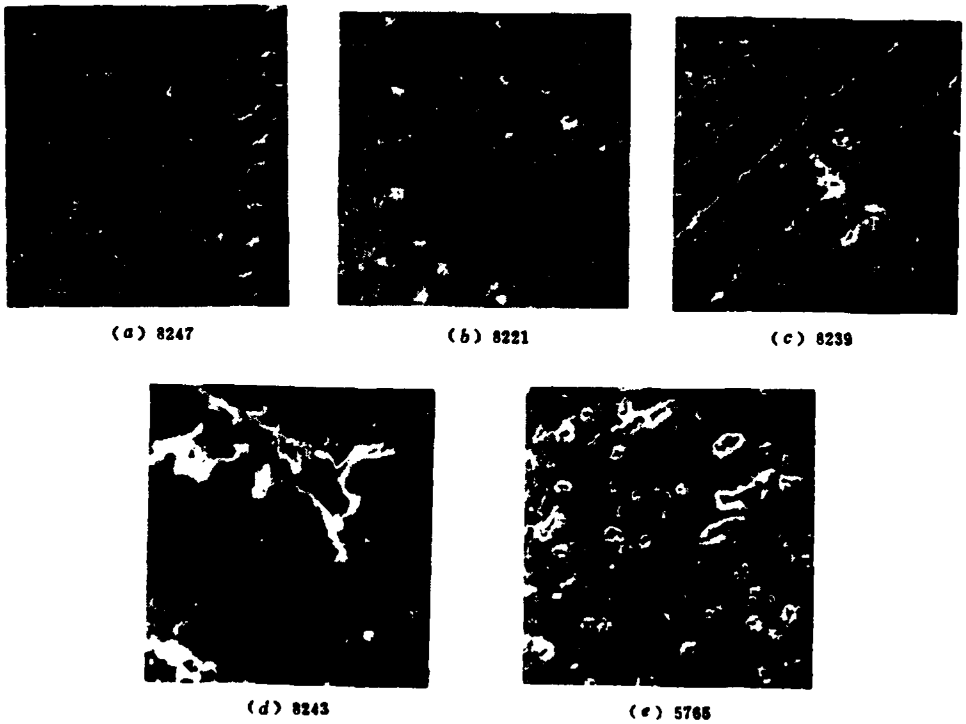


Fig. 1. Examples of Class and Directional Conditions of T Type Powder Grain Defects

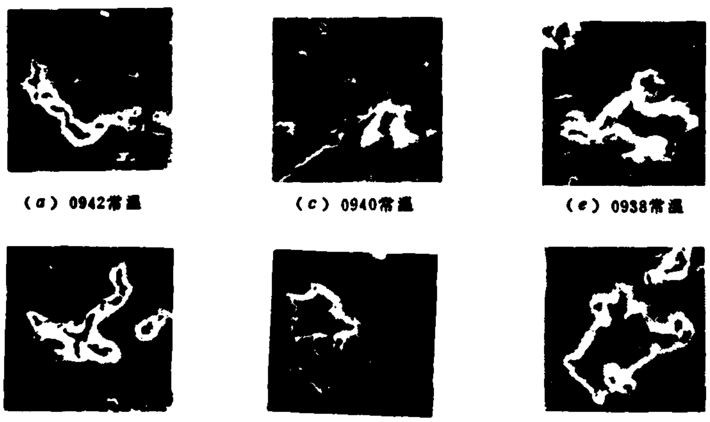
The second group uses conjugate sections of a similar group of powder grain (T type-A). After being kept under different temperatures, we observed the effects of temperature on the inherent defects (see table 2).

(1)序号	片(2)号	(3)样品批	(4)倍率	(5) [illegible]
a	0942	(6) T型-A	500×1/2	(12)常温保存，为0943的共轭面
b	0943	(7) T型-A	500×1/2	(13)0℃72小时后恢复常温
c	0940	(8) T型-A	500×1/2	(14)常温保存，为0941的共轭面
d	0941	(9) T型-A	500×1/2	(15)0℃72小时后恢复常温
e	0938	(10)T型-A	500×1/2	(16)常温保存，为0939的共轭面
f	0939	(11)T型-A	500×1/2	(17)0℃72小时后恢复常温

Table 2 The Effects of Temperature on Various Inherent Defects

Key: 1. Sequence number
2. Picture number
3. Specimen group
4. Multiplication rate
5. Temperature conditions experienced
6. T type-A
7. T type-A
8. T type-A
9. T type-A
10. T type-A
11. T type-A
12. Normal temperature kept on conjugate surface of 0943
13. After 72 hours at 0°C, normal temperature restored
14. Normal temperature kept on conjugate surface of 0941
15. After 72 hours at 0°C, normal temperature restored
16. Normal temperature kept on conjugate surface of 0939
17. After 72 hours at 0°C, normal temperature restored

See fig. 2 for pictures listed in table 2.



(a) 0942常温 (c) 0940常温 (e) 0938常温

a,c,e: normal temp.

(b) 0943低温 (d) 0941低温 (f) 0939低温

b,d,f: low temp.

Fig. 2. The Effects of Temperature on Different Shaped Defects

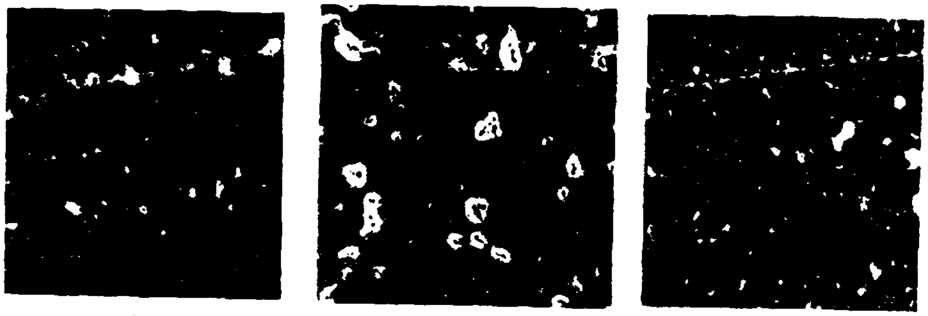
The third group. See table 3 for the micro-structures of T type powder grain manufactured under specific technological conditions.

(1) 序号	(2) 片号	(3) 样品批	(4) 取样部位	(5) 倍率	(6) 工艺特点
a	8155	(7) T型-C	(10) 中心	500×1/2	吸收棉烘干至 (13)
b	7720	(8) T型-C	(11) 非中心	500×1/2	水量含0.68% (14)
c	8177	(9) T型-D	(12) 中心	500×1/2	水量含3.0% (15)

Table 3 Examples of the Micro-Structures of Products Made With Specific Technology

Key:
1. Sequence number
2. Picture number
3. Specimen number
4. Sampling position
5. Multiplication rate
6. Technological characteristics
7. T type-C
8. T type-C
9. T type-D
10. Center
11. Not center
12. Center
13. Absorption cotton stoving
14. Water content is 0.68 percent
15. Water content is 3.0 percent

See fig. 3 for pictures listed in table 3.



(a) 8155 (b) 7720 (c) 8177

Fig. 3. Examples of the Structures of Products Made With Specific Technology

III. Results and Analysis

1. The Differences in Structural Defects of Two Groups of Specimens With Different Ballistic Behavior

A. The Ballistic Behavior Conditions of the Specimens

These two groups of T type powder grain were manufactured using the same directions and technological methods. The data determined from ballistic tests is listed in table 4.

(1) 批号	(2) 温度/℃	(3) 装药量/克	(4) 膛压 P_m/千克·厘米$^{-2}$	$\frac{P_m^{-40} - P_m^{15}}{P_m^{15}}$
(5) T型-A	15℃	112	3156	(9) +9.2%(反常)
(6) T型-A	-40℃	112	3447	
(7) T型-B	15℃	109	3125	(10) -6.4%(正常)
(8) T型-B	-40℃	109	2926	

Table 4 Examples of Data on the Ballistic Behaviors of T Type-A T Type-B

Key: 1. Group number

2. Temperature/degrees centigrade
3. Powder charge/gram
4. Bore pressure Pm/kilogram · centimeter^2
5. T type-A
6. T type-A
7. T type-B
8. T type-B
9. Abnormal
10. Normal

B. Selection of Multiplication Rate

The sizes of the powder grain microdefect holes are not equal nor are their shapes the same. Given that the defects are not directly observed, the defect apertures generally do not exceed 100 micron. It has been practically proven that if there are no external defects, then the apertures of the micro-defects inside the powder grain rarely exceed 40 micron. It is estimated that defects with many holes and large apertures should have great effects on ballistic behavior. In order to adapt to these observation goals and make it convenient to carry out statistical contrasts for different groups, we selected a fixed observation multiplication rate of 500. In this way, the observation area was 0.2x0.2 square millimeters. We could see everything on the kinescope when this area had a hole with a 1 millimeter aperture. There was also a clear and definite trace on the picture. The picture's recording multiplication rate was 500x½.

C. Method of Showing the Class of the T Type Powder Grain Structure

For the class of the defect condition, we used the size of a single hole's section exposed on the observed surface as a basis for measurement. Because the forms are not uniform, it is difficult to give accurate statistics and so we temporarily divide the size of the defect sections into four classes.

Class I structure. The powder is compact and there are very small defects. If a 500x½ multiple picture is directly observed, there are no visible holes or cracks. They are called "without holes" for short. Thus, in the observed area, no holes above 1 millimeter appear; for example, picture 8247 (see fig. 1a). The veins in the picture are marks left from knife cuts but no obvious cracks or holes are visible.

Class II structure. The powder is relatively compact and there are only a scattering of small defects. The radial size of these holes is less than 10 micron. On a 500x½ picture, the aperture does not exceed 2.5 millimeters and thus they are included in the class II structure. They are called "small holes" for short. See for example picture 8221 (fig. 1b).

Class III structure. The powder is not sufficiently compact and in the observed areas the size of a single hole aperture is in the range of 10 to 12 micron. On a 500x½ multiple picture, the aperture is in the range of 2.5 to 5 millimeters. If there is no medium hole but many small holes concentrated together forming a pile, then the area extension is quite large reaching 40x40 micron^2 (1x1 centimeter^2 area on the picture); ot the small holes are adjacent and strung together forming a continuous crack with a length reaching to 100 micron (its length reaches to about 2.5 centimeters in the picture). This also belongs to the class III structure ans is called the "medium hole" for short. See for example picture 8239 (fig. 1c).

Class IV structure. The powder is not compact. In the observed area, the aperture reaches to a relatively large hole of 20-40 micron (a 5-10 millimeter aperture in the picture) or two "medium holes" concentrated together which have a linking tendency. This belongs to the class IV structure and is called

the "large hole" for short. See for example picture 8243 (fig. 1d).

Statistics were not done on the classes of the light area defects. It was only observed whether or not the cracks had directional tendencies. See for example picture 5765 (fig. 1e).

D. The Structural Differences of Two Groups of Powder Grain

The T type-A and T type-B group powder grains with different ballistic behaviors were extracted from the center section and statistics were done according to the four classes. The proportion of the pictures occupied by light surface sections with directional tendencies as well as the statistical conditions are listed in table 5.

(1) 批号	(2) 弹道性能	(3) 药粒中心切面缺陷情况				(4) 浅表切面定向比例
		I	II	III	IV	
(5) T型-A	(7) 反常	0	0.60	0.30	0.10	5/9
(6) T型-B	(8) 正常	0.10	0.55	0.30	0.05	4/10

Table 5 Micro-Structural Conditions of the T Type-A and T Type-B Groups

Key:
1. Group number
2. Ballistic behavior
3. Defect conditions of powder grain in center section
4. Directional ratio of light surface section
5. T type-A
6. T type-B
7. Abnormal
8. Normal

It can be seen from table 5 that in the ratio of the abnormal group and normal group of ballistic behavior, the defects of the former are more serious than those of the latter. This is shown in that for the normal group, the probability of the appearance of large holes is greater than that for the normal group and the probability of "no holes" in the normal group is greater than for the abnormal group. Aside from this, there was a greater directional tendency of defects which appeared in the light surface of the abnormal group. These results presented a certain revelation: under normal temperature, the inherent microdefects in powder grain can have a specific effect on low temperature ballistic behavior. If the defect holes or cracks are larger, greater in number or have strong directional tendencies, then they will reach a certain level wherein they can cause ballistic behavior to worsen.

2. The Effects of Low Temperature Storage on the Microdefects of Powder Grain

In order to clarify the way the inherent microdefects affect low temperature ballistic behavior, it is necessary to carry out direct observations of microdefects while they are changing at declining temperatures. A T type-A group powder grain is cut open cross-wise, a corresponding position is taken on the conjugate surface and one piece is kept at room temperature; after the conjugate surface is kept at a low temperature (0°C for 72 hours), it returns to a normal temperature, and two pieces are observed for comparison under similar conditions. It can be seen from the results shown in table 2 and from fig. 2 that different shaped defects are effected by temperature. A detailed explanation is given below.

Continuous cracks: pictures 0942 and 0943 are a group of conjugate surfaces on T type-A powder grain. Pictures of

corresponding points on the conjugate surfaces are mirror images of eachother. Because temperature conditions are different there are slight changes. If the two pictures are placed together and the knife mark directions are mutually symmetrical, from the outline of the crack shaped defect stretching to the bend, we can discriminate the traces of the mirror image. For example, as shown in fig. 4, the following three points are worthy of attention: 1) the crack shaped defects having undergone low temperature processing but not yet having produced extensive spreading can be explained from the basic symmetrical mirror image assumed by points A and A' in fig. 4. 2) If, in the proximity of the crack, there is no connecting defect while under normal temperature, after low temperature processing there can be mutual linkage. This will cause the ends of the crack to stretch or appear branched. This can be shown by contrasting areas B and B' in Fig. 4. 3) Some of the scattered small holes with diameters less than 2 micron shrink or disappear after low temperature processing. This is possibly due to the quadratic effects of the temperature restoring process.

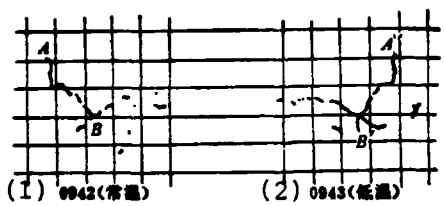


Fig. 4. Crack Defects Following Temperature Changes

Key: 1. Normal temperature
2. Low temperature

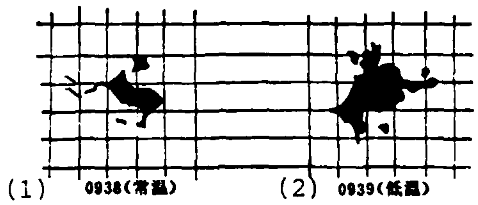


Fig. 5. Adjacent Hole Defects Following Temperature Changes

Key: 1. Normal temperature
2. Low temperature

Single holes: pictures 0940 and 0941 are a set of conjugate surfaces. Under normal temperature, the photo area of a single hole is about 1.6x1.5 millimeter^2, the outline of the hole is close to a quadrilateral and the lengths of the sides are about 6 micron. After low temperature processing, the holes appear enlarged, and each side seems to be 3 times larger.

Several adjacent holes: pictures 0938 and 0939 are a set of conjugate surfaces. Here, there are several hole defects adjacent to eachother as shown in fig. 5. Under normal temperature, after a flat hole (with a length of 9 millimeters and a width of approximately 3 millimeters) undergoes low temperature processing, it becomes a defect with a length of about 13 millimeters and a width of about 7 millimeters. Moreover, the small holes originally nearby expand and at the same time link up with it.

The changes of the above mentioned defects are not produced

by external forces but are the results of the powder grains shrinking out of shape when their own static temperature declines. The progressive change patterns of the structural defects of powder grains with declining temperatures is: the cracks branch, the flat holes round out, the adjacent holes link together and the single holes enlarge.

When we consider how different the defects exposed on the cut surface are from the specific conditions of the temperature changes within the powder grain bodies, the attained pattern can only specifically explain the tendencies of the defect changes.

3. The Mechanism of Inherent Defects of Abnormal Ballistic Behavior for T Type-A Group Propellant

During the combustion process in the bore, the powder grain undergoes an uneven chemical reaction on the shared surface. Under certain conditions, the larger the surface area of the reaction, the larger the speed of forming the resulting gas. The area of the produced reaction surface is made up of two parts; the exposed surface determined from the structural defects and the combustion surface determined from the powder type. The size of the exposed surface is difficult to measure in gun-powder design and it is also not easy to control it visually during production. Because of this, each group of products necessarily fluctuates in ballistic behavior data due to the instability of the structure's inherent defects.

If the reaction area is maintained unchanged, the linear speed of the combustion surface thrust declines in accordance with the powder temperature decreases, and it increases with the rises of the resulting gas pressure. The bore pressure is

the combined result of the reaction surface and the effects of the powder temperature. Therefore, the development of the structural defects and the effects of the low temperature on the reaction are mutually contradictory factors. When the powder grain has lighter (small, scarce, scattered) defects under normal temperature, under low temperature even though the defects can cause the surface area of the reaction to enlarge, yet the effect of the expanded surface on the generating speed of the gas product is not sufficient to compensate for the low temperature's inhibitory effect on the reaction. When the other conditions are constant, then the low temperature bore pressure cannot be higher than normal temperature data. The T type-B group possibly belongs to this category.

If, under normal temperature, the inherent defects of the powder grain structure are serious (large, numerous, dense and with directional tendency strength), when the temperature drops, not only do the small holes enlarge but at the same time the adjacent holes link up, the cracks branch out, and the directional extensions cause the combustion surface to rapidly enlarge. When they expand to a critical degree so that the effects of the area factor on the generating speed of the gas product exceeds the inhibitory effects caused by the temperature lowering, the pressure in the bore develops rapidly and there is the "abnormal" phenomenon of the low temperature bore pressure being higher than the normal temperature data. This T type-A group is possibly an example of this.

On the other hand, when the powder is compact and there is a tendency towards not having defects, it can be supposed that under low temperature, because the defects cause the reaction area to enlarge, there is also a tendency towards not having defects. As regards this type of specifically manufactured

homogeneous propellant, the low temperature bore pressure is necessarily smaller than the normal temperature data. Given controlled and constant test conditions, under specific temperatures, the numerical values should be lower than for data on powder grain with defects. There also tends to be a certain limiting value.

4. The Physical Chemistry Principle of Forming Inherent Defects in Powder Grain

T type powder grain is manufactured by using a composite possessed of moisture absorbing cotton, is gelatinized by a repellant solvent (acetic ester), and after being shaped by thrusting, it undergoes salting-out dehydration, heat drives out the solvent and there is finally drying. Here we will only discuss the portion related to the formation of defects.

Acetic ester and water are partially soluble liquids. Their solubility curve is shown in fig. 6 [6].

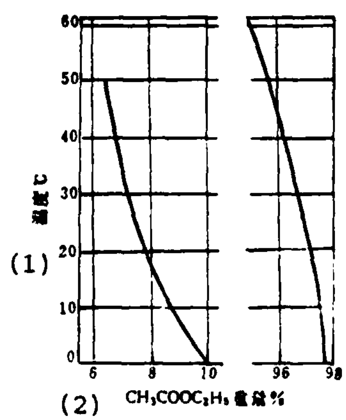


Fig. 6. The Solubility Curve of Acetic Ester and Water

Key: 1. Temperature (degrees centigrade)
2. Weight

The special feature of the figure is that when the temperature rises, the ester contents in the two saturated solvents both decline. A saturated solvent which takes water as the main ingredient and which cannot dissolve Nc is also called the "water phase"; a saturated solvent which takes ester as the main ingredient and has a certain dissolving power for Nc is called the "ester phase". The ester phase can have priority wetting for Nc wherein the water on the surface of the Nc will be expelled and form liquid drops. After mixing the materials, the liquid drops concentrate together to form a larger water phase area, a portion of which can be eliminated along the cracks of the material. The remaining water phase which has not been eliminated is then suspended and distributed in a sticky and dense material made up of Nc and the solvent. During gelatinization and thrusting, irregular bending occurs in accordance with the rheological conditions and becomes the "water phase area" scattered in the powder grain. When the powder grain is heated in a salt solution, the acetic ester and water molecules move and bring about a change in the composition near the surface which causes local Nc condensation. When a very small water phase area has rises in temperature, it can completely dissove into an ester phase, and after the powder grain is manufactured, there is the possibility of fluctuation in the local properties but no apparent defects are formed. Some larger water phase areas have partial dissolving into the ester phase when there is rising temperature but there is still a remaining water phase. The Nc all around it condenses and after reaching common boiling points, the consistency of the ester phase enlarges in accordance with the evaporation. The powder cannot promptly fill the spaces in the powder grain and

and during the process wherein the solvent and water are eliminated, microdefects are formed in the powder grain.

In accordance with this analysis, if we are able to effectively lower the water content of the gelatinized powder and cause $H_2O:CH_3COOC_2H_5 \ngtr 3.5:96.6$ (weight ratio), then there should be a great improvement of the powder grains inherent defects.

5. The Structures of Specially Manufactured Products and the Results of Ballistic Behavior

The insufficient elimination of water during gelatinization is the major reason for the production of inherent defects and when the level of inherent defects becomes serious then this can possibly cause abnormal ballistic behavior. To test whether or not this judgement is correct, we used a specially manufactured absorbing cotton which effectively lowered the remaining quantity of water. After manufacturing the powder grain, we then measured the micro-structure and ballistic behavior. The results are shown in table 6.

(1) 批号	(2) 吸收棉含残余水量	(3) 药粒中心缺陷情况				(8) 弹道性能情况			
		(4) I级	(5) II级	(6) III级	(7) IV级	(9) 温度	(10) 装药量	(11) 膛压	$\Delta P_m/P_m^{15°}$
T型-C (12)	0.68%	0	0.90	0.10	0	15°	(14) 114克	3146	-12.8%
						-40°	(15) 114克	2658	
T型-D (13)	3.0%	0	0.90	0.10	0	15	(16) 114克	3084	-11.2%
						-40°	(17) 114克	2636	

Table 6 The Structure and Ballistic Behavior of Specially Manufactured Products

Key:
1. Group number
2. Amount of remaining water contained in absorbing cotton
3. Powder grain central defects
4. Class I
5. Class II
6. Class III
7. Class IV
8. Ballistic behavior
9. Temperature
10. Amount of powder
11. Bore pressure
12. T type-C
13. T type-D
14. 114 grams
15. 114 grams
16. 114 grams
17. 114 grams

When we compare the data of table 6 with that of tables 4 and 5, we can see that if specially manufactured products rigorously control the remaining water, then the defects in the powder grain will be small and evenly distributed. Table 3 and fig. 3 are typical examples of the micro-structures of T type-C and T type-D specially manufactured powder grain. It was practically proven that by controlling moisture we can effectively control the quality of the powder grain structure and thus attain normal ballistic behavior.

Conclusion

The observations of T type powder grain by means of a scanning electron microscope and the results of preliminary statistics presented in this article show that the inherent defects of powder grain have a certain effect on ballistic behavior. If during a combined specific production process, we can grasp the key strengthening of administration so that we improve the micro-structures of the products and avoid abnormal

ballistic behavior, this will be quite satisfactory.

The scanning micro-pictures presented in this article were taken by Dang Guangyue and Dong Yuying to whom we would like to express our gratitude.

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